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PATENT SPECIFICATION 954,613

DRAWINGS ATTACHED.

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COMPLETE SPECIFICATION.

Improvements in or relating to Hot Pressing.

We, THE BRITISH ALUMINIUM COMPANY LIMITED, a Company registered under the laws of Great Britain, of Norfolk House, St. James's Square, London, S.W.1, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to improvements in hot pressing and is particularly concerned with the hot pressing of refractory hard metal material powders in a graphite or carbon die, the hot pressing operation being performed at temperatures of 1000° C. or higher.

As used herein in the Specification and claims the expression "refractory hard metal material" refers to material which possesses a low electrical resistivity, a low solubility in molten aluminium and molten electrolyte under cell operating conditions, is wettable by molten aluminium under cell operating conditions and has good stability under conditions existing at the cathode of a reduction cell. The preferred refractory hard metal material for at least that portion of the surface of such element in contact with the molten aluminium consists essentially of at least one of the materials selected from the group consisting of the carbides and borides of titanium, zirconium, tantalum and niobium, and mixtures thereof, such materials being found to exhibit all or substantially all of the above properties.

The expression "consists essentially" as used hereinafter in the Specification and the claims means that the refractory hard metal material does not contain other substances in amounts sufficient to materially affect the desirable characteristics of the material, although other substances may be present in minor amounts which do not materially affect such desirable characteristics, for example, small proportions of oxygen, nitrogen and titanium nitride in titanium boride.

The relatively recent discovery that refractory hard metal materials perform exceptionally well as current-conducting elements in electrolytic cells for the production of aluminium has prompted considerable activity in the application of powder metallurgical techniques to refractory hard metal materials. Powder pressing and sintering either subsequently or simultaneously have proved to be the only practical method of obtaining a coherent metallic mass of these materials but difficulties with the dies significantly affect hot pressing production.

In hot pressing, the mould in which the powder is pressed must be able to withstand the high pressures in the range of 500—5000 p.s.i. or even substantially higher pressures for sustained periods of time while the powder is sintering. The sintering of the compressed object is carried out at times and temperatures depending upon the powder material.

In view of the high temperatures involved in all hot pressing operations the choice of mould materials is narrowed considerably. Dies for use in high temperature hot-pressing processes which are performed at temperatures above 1000° C., and usually from above 1500° C. to about 2300° C. are commonly made of graphite in view of the resistance of this material to the high temperatures and pressures involved. In some cases, however, the material to be hot-pressed may be one which either reacts with or is mutually soluble in the material of the die at the pressing temperature, and in these cases

there is a tendency for the hot pressed body to stick to the die. The sticking may necessitate that a new die be used for each pressing operation thereby adding considerably to the cost, or the sticking may cause the die to break before the pressing operation is completed. Sometimes the pressed body will break during cooling as a result of the differential contraction of the die and the body.

Another problem encountered is the narrowing of the die at the extremities in relation to the centre. In such cases the die has a larger cross-sectional area at the centre than at the ends. This is due to the often-overlooked factor of elasticity of graphite. When the inside surface of the die takes on this concave appearance due to the great pressures applied in hot pressing, there is an increased tendency for the hot-pressed body to crack the die on ejection from the die cavity after pressing.

As an example of the problems encountered when hot-pressing refractory hard metal materials in carbon or graphite dies, when titanium diboride is hot-pressed to bar form in a graphite die at a temperature of the order of 2000° C. there is a marked tendency for the titanium diboride to stick to the die with the result that the die breaks before the pressing operation is completed or the pressed body of titanium diboride breaks during cooling as a result of the differential contraction of the die and the pressed bodies. These difficulties are not encountered when the hot-pressing operation is performed at temperatures below 1700° C. but it is not possible to produce a satisfactory strong and dense titanium diboride body at such low temeperatures. It is thought that titanium diboride and carbon have some mutual solubility at temperatures of the order of 2000° C. or above, or that the effect may be due to small amounts of impurity such as boric oxide (B_2O_3) which is not economic to remove from the titanium diboride powder. Whatever the reason for the sticking effect, it has been found that titanium diboride may be satisfactorily hot-pressed at temperatures of the order of 2000° C. and above in a graphite die when the wall surfaces defining the cavity in the die are periodically ground and polished smooth to remove "pits" in the die surface and to overcome the problems encountered due to the elasticity of carbon.

In addition to the above, after use, the interior finish of the die becomes roughened sometimes due to chemical attack, and sometimes because of gouging by projections on the surface of the titanium diboride bar. The projections are the result of the refractory hard metal material of the bar being forced into pits that are inherently present in the graphite stock. As pressure is applied during hot pressing the refractory hard metal powder is forced into the pits in the graphite surface and tends to enlarge these cavities increasing the friction between the refractory hard metal material and the surface of the graphite die. Furthermore, the refractory hard metal material becoming lodged in the cavities of the graphite surface, adds to the stresses imposed on the graphite die during ejection of the finished pressed refractory hard metal body. This interlocking effect between the refractory hard metal material and the graphite die further increases the tendency of the die to break upon ejection of the refractory hard metal body.

It is an object of the present invention to provide an improved method of hot pressing refractory hard metal powders whereby the disadvantages referred to shall be materially reduced.

According to the present invention there is provided in the method of hot pressing refractory hard metal material powders in the die cavity of a graphite die, the step of grinding the die cavity surface to polish the surface thereby increasing the usable life of the die and thereafter charging the die with the powder and subjecting the powder to a hot pressing operation.

Before the present invention the number of pressings possible using one graphite die was usually no more than from one to three. The maximum that had been attained was five pressings of refractory hard metal material in one graphite die. By utilizing the present invention, the number of pressings can be increased up to as many of twenty-one pressings before a die failure occurs. It has generally been found that from one to three pressings can be made before regrinding is necessary. Usually only a few minutes of grinding is required to restore the die to a satisfactory condition. However, the amount of grinding and corresponding time required depend upon the severity of die attack and general condition of the die.

As an illustration of the increase in die life and the great savings possible by the application of the present invention, the following data presented in Table I is of particular interest. Ninety-two bars were pressed in fourteen dies; seven dies which were not treated according to the invention failed with an average of 2.7 pressings per die. Three of these dies were lost because of die attack and four dies broke on removal of the pressed bar.

TABLE I.

Die	No. of Pressings	Remarks
1	2	Heavy die attack
2	3	Die cracked on removal
3	1	Die cracked on removal
4	5	Die attack
5	2	Die attack
6	2	Bar stuck in die
7	4	Die broke on removal of bar
	Average pressings per die. = 2.7	
8	11	Die honed
9	6	Die honed
10	10	Die honed
11	6	Die honed
12	9	Die honed
13	10	Die honed
14	21	Die honed
	Average pressings per die = 10.4	

By periodically grinding the die after several pressings, the useful life of the die is greatly increased. The average die life can be increased from 2.7 pressings per die obtained without practising the invention to 10.7 pressings per die by treating the die according to the invention. As shown in Table I as many as twenty-one pressings are obtainable in one graphite die by periodic grinding according to the invention.

In the accompanying drawings are illustrated the equipment for pressing refractory hard metal powder into bars and one embodiment of the invention.

Fig. 1 is an elevation view in section of a furnace used in hot pressing refractory hard metal powder, including the die and accessories.

Fig. 2 is a vertical elevation view in section showing one embodiment of the die ground slightly oversize at the bar-removal end to facilitate the ejection of the bar out of the die.

The hot pressing furnace assembly as shown in Fig. 1 generally comprises a furnace shell 10, a tubular graphite insulating sheath 14, a tubular graphite resistor heater tube 16, and elongated tubular graphite die 22. Furnace shell 10 is of stainless steel (or mild steel) with lids or cover plates 11 and contains lampblack insulation 12 immediately within and extending inwardly to the graphite sheath 14, which is spaced from resistor heater tube 16, also of graphite, by ceramic rings 18 leaving an additional insulating gas space 19. The heater 16 is electrically connected to power supplying terminal blocks 30 and is insulated from the stainless steel lid or cover plate 11 by discs 34 of any suitable insulating material, such as "Transite" (a material made from asbestos and Portland cement and sold by Johns-Manville Sales Corporation, of New York, N.Y.) or "Teflon" (a plastic consisting of tetrafluoroethylene polymer and sold by E. I. du Pont De Nemours & Co., of Wilmington, Del). The words "Transite" and "Teflon" are Registered Trade Marks. The heater 16 is electrically insulated from the die 22 at the top and bottom by Transite packing flanges 24 which are packed to form a gas seal with the elongated open tubular graphite die 22. Water cooled copper rings 32 are installed in contact with each end of the die to maintain these ends below oxidation temperature during the hot pressing cycle. Good contact for thermal conductivity is assured since the lower ring supports the weight of the die and the upper copper ring is held in contact by a spring loaded plate (not shown).

Access for optical pyrometry is provided through a sight assembly 40 hermetically attached to the furnace shell surface. The assembly contains glass ports 42 for viewing into sight tubes 44. Sight tubes 44 which may be of graphite, extend through openings in the shell 10 and are axially disposed with openings in the sheath and heater tube for viewing the graphite die and to provide access to gas spaces 19 and 21. Purge gas entry ports 46 may be provided in the sight assembly as a convenient means for introducing purge gas into the furnace assembly or purge gas can be introduced through an inlet 48 provided in the lower flange 24A. The gas after entering through inlets 46 passes through sight tubes 44 into gas space zones 19 and 21. Openings are provided in the stainless steel lids or covers 11 as exits for the purge gas circulating through the hot pressing furnace assembly.

Figure 2 is an exaggerated illustration of the embodiment wherein the top end of the die 22 is ground slightly oversize to counteract the effects encountered by virtue of the tendency of the graphite die to bulge in the central one-third portion under pressure during hot pressing. When the die is ground in this manner the pressure required to extract the bar is relieved progressively as the bar is moved outward from within the graphite die.

The grinding of the die may be accomplished in any suitable manner, for example, by clamping the graphite die against a vertical pole which supports the die in a fixed position. A suitable grinding machine (such as Sunnen Hone Model An-112 manufactured by the Sunnen Products Company, St. Louis, Mo.) is secured above the die and attached to the vertical pole so as to be easily guided into and through the bore of the graphite die. The motor driven grinder may be either manually or mechanically inserted into the bore and caused to abrade the surface of the graphite. The operation is very flexible and any depth

of cut necessary to remove all the blemishes on the graphite surface can be accomplished by the operator. Generally, from 0.020"—0.040" of die surface is removed.

As indicated above, dies have failed frequently during pressing with resulting damage to the pressed piece. The die wall undergoes some chemical attack during the pressing resulting in additional cavities or pits in the surface of the graphite die. Further, the inside diameter of the centre one-third of the die increases up to 0.003 inch per pressing as a result of the elasticity of the graphite die material. Thus the centre of the refractory hard metal material bars produced increases in diameter with continued use of the die. The continued use of the die which has suffered corrosion pitting and localized expansion also puts increasing strain on the thin walled and uncorroded end sections as the pressed pieces are being removed. Grinding the die after a few pressings polishes the inner surface of the graphite die thereby significantly eliminating the pits and blemishes in the surface. The grinding operation removes a small portion of the surface of the graphite die providing a bore of uniform diameter.

In addition to grinding of the die in a manner to produce a uniform diameter bore, techniques have been developed for grinding the bar-removal end of a die to facilitate ejection thereof as shown in Figure 2. In this embodiment the top (or bottom) end of the die is ground slightly oversize in such a manner that when the bar is extracted the pressure on the die is relieved progressively as the bar is moved. This embodiment avoids any difficulty encountered during the extraction of the bar from the die wherein the bar may become tight as it is moved into the upper or lower undeformed portion of the die which sometimes causes rupture of the die. Alternatively, the die may be ground slightly "bell mouthed" so that the bar can be removed still more easily.

WHAT WE CLAIM IS:—

1. In the method of hot pressing refractory hard metal material powders as defined herein in the die cavity of a graphite die, the step of grinding the die cavity surface to polish the surface thereby increasing the usable life of the die and thereafter charging the die with the powder and subjecting the powder to a hot pressing operation.

2. A method according to Claim 1 wherein the die cavity is of elongated tubular form.

3. A method according to Claim 2 wherein the die cavity is ground in such a manner as to provide a greater cross-sectional area at one end thereof than that of the remainder of the cavity.

4. A method according to Claim 1 or 2 wherein the die cavity is ground periodically between hot pressing cycles of from one to three hot pressing operations.

5. A method according to any one of the preceding claims wherein the refractory hard metal material consists essentially of at least one of the materials titanium diboride and titanium carbide.

6. A method of hot pressing refractory hard metal material powders substantially as herein described.

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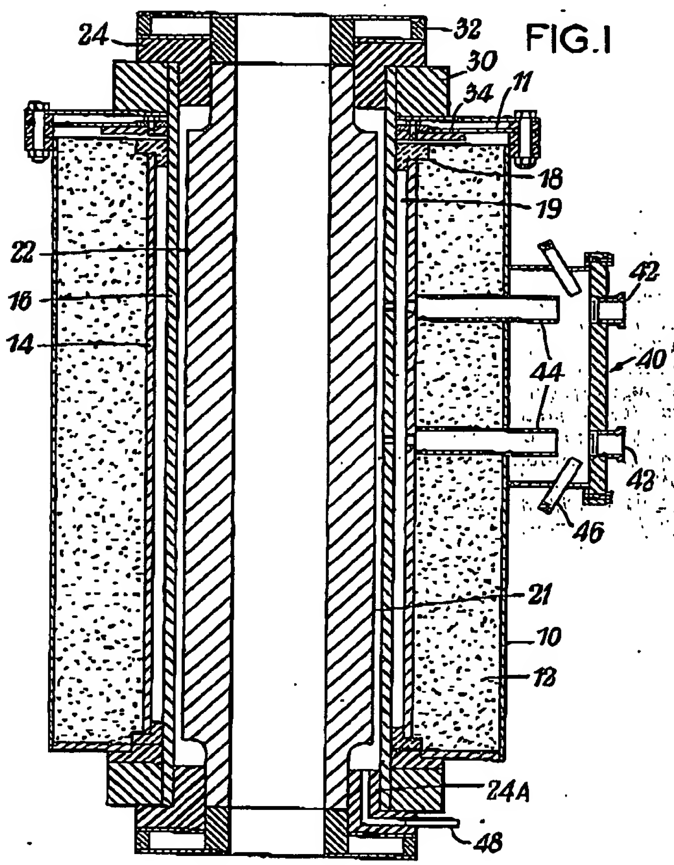
954613 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*

FIG.1



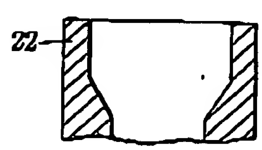


FIG.2